AD-A146 233 TIME-TEMPERATURE DEPENDENCE OF THE STRENGTH OF COMMERCIAL ZIRCONIA CERAMICS(U) ARMY MATERIALS AND MECHANICS RESEARCH CENTER WATERTOWN MA 1/1

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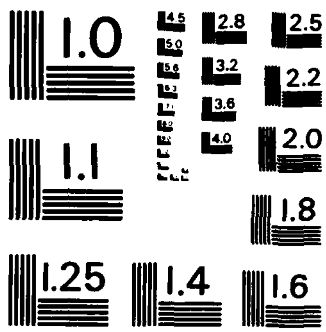
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TIME-TEMPERATURE DEPENDENCE OF THE STRENGTH OF COMMERCIAL ZIRCONIA CERAMICS

LISELOTTE J. SCHIOLER, GEORGE D. QUINN and R. NATHAN KATZ

April 1984

Prepared under
Interagency Agreement DE-AE-101-77 CS51017

by
ARMY MATERIALS AND MECHANICS RESEARCH CENTER
WATERTOWN, MASSACHUSETTS 02172

U.S. DEPARTMENT OF ENERGY

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER AMMRC TR 84-16	2. GOVT ACCESSION NO. AD-A146 233	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) TIME-TEMPERATURE DEPENDENCE OF THE STRENGTH OF COMMERCIAL ZIRCONIA CERAMICS		5. TYPE OF REPORT & PERIOD COVERED 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Liselotte J. Schioler, George D. Quinn, and R. Nathan Katz		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Army Materials and Mechanics Research Center Watertown, Massachusetts 02172 DRXMR-MC		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Interagency Agreement DE-AE-101-77 CS51017 AMCMS Code: 69200R.8891
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Department of Energy Office of Vehicle & Engine R&D, Conservation and Renewable Energy, Washington, DC 20545		12. REPORT DATE April 1984 13. NUMBER OF PAGES 11
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES Presented at the DoE Contractors' Meeting, Dearborn, Michigan, November 1983. Published in the Proceedings of the Twenty-First Automotive Technology Development Contractors' Coordination Meeting, Society of Automotive Engineers, Inc., Warrendale, PA March 1984.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Zirconium oxides
Heat engines
Mechanical properties
Ceramic materials
High temperature

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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ABSTRACT

The unusual combination of attractive properties exhibited by transformation toughened zirconias (TTZ's) has focused attention on them as leading candidates for application in the adiabatic Diesel engine. These materials are age-hardened ceramic alloy systems and as such they are likely to be susceptible to overaging and loss of strength at long times at high temperatures. This paper presents preliminary data on the microstructural, phase and dimensional stability of aged TTZ's together with some data on the effects of aging on strength, toughness and modulus. Stress rupture data on TTZ's with both MgO and Y_2O_3 additions show a considerable decrease in load carrying ability at temperatures of 800°C and above.

A-1

Time-Temperature Dependence of the Strength of Commercial Zirconia Ceramics

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ABSTRACT

The unusual combination of attractive properties exhibited by transformation toughened zirconias (TTZ's) has focused attention on them as leading candidates for application in the adiabatic Diesel engine. These materials are age-hardened ceramic alloy systems and as such they are likely to be susceptible to overaging and loss of strength at long times at high temperatures. This paper presents preliminary data on the microstructural, phase and dimensional stability of aged TTZ's together with some data on the effects of aging on strength, toughness and modulus. Stress rupture data on TTZ's with both MgO and Y_2O_3 additions show a considerable decrease in load carrying ability at temperatures of 800°C and above.

BECAUSE OF THEIR UNUSUAL combination of properties (E ~ steel, k ~ 1/5 to 1/50 that of Si_3N_4 or SiC, an α ~ steel, MOR ~ 100 KSI, and a high K_{Ic} ~ 10), transformation toughened zirconias (TTZ) are leading candidates for cylinder liners, piston caps, head plates, valve seats, and other components for the adiabatic Diesel engine. These materials are age-hardened ceramic alloy systems and as such they are likely to be susceptible to overaging and loss of strength at long times at high temperatures (i.e. close to the age hardening temperatures). The possibility of overaging with its likely negative impact on materials performance was identified as a critical area of ignorance in the preliminary technology assessment on ceramics for Diesel engines previously prepared by AMMRC (1). Accordingly, a task was initiated to: a) define the extent and magnitude of the overaging (if any), and b) develop toughened ceramic alloy systems which would not be susceptible to overaging at temperatures which may be encountered in advanced Diesels (1100-1200°C). Figure 1 gives an overview of the objective and approach of these two tasks. This paper will review progress on the task being carried out in-house at AMMRC. Professor Tien's paper later in this session (2) will review progress on the second task, being conducted at the University of Michigan.

TOUGHENED CERAMICS

SUBTASK I - AGING STUDIES OF TTZ

P.I.: L. SCHIOLER - AMMRC
OBJECTIVE: *DEFINE EXTENT OF AGING PHENOMENA, IF ANY*
APPROACH: PHASE I - EXPOSE COUPONS FOR VARIOUS t-T AND EVALUATE CHANGES IN:
- PHASE CONTENT - DENSITY
- MICROSTRUCTURE - K_{IC}
- DIMENSIONS - E

PHASE II - EVALUATE MOR AND FAILURE MODE AFTER EXPOSURE

SUBTASK II - TOUGHENED Al_2O_3 BASED CERAMICS

P.I.: PROF. TIEN - UNIVERSITY OF MICHIGAN
OBJECTIVE: DEVELOP OVERAGING RESISTANT ALLOY SYSTEMS (UP TO 1200°C)
APPROACH: SCREEN Al_2O_3/Cr_2O_3 - ZrO_2/HfO_2 ALLOY SYSTEMS
SCREEN MULLITE - ZrO_2/HfO_2 ALLOY SYSTEMS
EVALUATE IN TWO PHASES AS IN SUBTASK I

Figure 1.

Zirconia ceramics of interest for Diesel engine application include transformation toughened zirconia (TTZ), partially stabilized zirconia (PSZ), and fully stabilized zirconia (FSZ). Figure 2 summarizes the main microstructural differences among these three types of zirconias and provides typical room temperature mechanical properties. The TTZ's are of major interest for application in highly stressed engine components due to their relatively high strength and

toughness. The high strength and toughness of TTZ is largely a result of the martensitic transformation of the metastable tetragonal phase to the stable monoclinic phase under stress. However, long time exposure to high temperatures may result in growth of the metastable tetragonal phase which in turn can trigger spontaneous transformation to the monoclinic phase, with the potential of signficant reductions in strength and toughness. Therefore, this paper will focus on our studies of the effect of aging on the physical and mechanical properties of TTZ's. Aging studies were carried out both with and without an applied load on the specimen. In the former case, density, phase content, microstructural stability, retained RT modulus of rupture (MOR), fracture toughness K_{Ic} and modulus of elasticity (MOE) were measured. In the latter case, stepped temperature stress rupture (STSR) and supplemental stress rupture (SR) tests were conducted. From these studies it is possible to define the temperatures and stress levels below which TTZ's can be used without severe instability.

ZIRCONIA CERAMICS

TRANSFORMATION TOUGHENED (TTZ's)	•PARTIALLY STABILIZED WITH Y_2O_3, MgO, OR CaC •METASTABLE TETRAGONAL PHASE •OVERAGING BY TETRAG → MONO	MOR ≈ 500-600 MPa K_{IC} ≈ 8-10 $MN/m^{3/2}$
PARTIALLY STABILIZED (PSZ's)	•PARTIALLY STABILIZED WITH Y_2O_3, MgO, OR CaO •NO METASTABLE TETRAGONAL PHASE	MOR ≈ 300 MPa K_{IC} ≈ 4 $MN/m^{3/2}$
FULLY STABILIZED	•STABILIZED WITH Y_2O_3, MgO, OR CaO •ALL CUBIC PHASE	MOR ≈ 200-250 MPa K_{IC} ≈ 3 $MN/m^{3/2}$

Figure 2.

MATERIALS

The materials studied were commerically available zirconias that were supplied free of charge by the manufacturers or Cummins Engine Company (material N only) on the condition that they be given code letters. Materials C, MN and N were optimally aged when received, while material F was not (the MOR and K_{Ic} actually increased after heat treatment at 1000°C for 100 hours, but then decreased upon further heat treatment). The bend specimens were machined such that all samples had the same surface finish. This was done to minimize the effect of the surface transformation layer on comparison of the properties. As received properties of these four TTZ's are shown in Figure 3.

AS-RECEIVED PROPERTIES OF COMMERCIAL TTZ

CODE	ADDITIVE	DENSITY (g/cc)	RT MOR (MPa)	K_{IC} ($MN/m^{3/2}$)	RT MOE (GPa)
C	MgO	5.67	592	7.9	227
F	MgO	5.51	447	5.3	215
MN	MgO	5.65	520	10.2	199
N	Y_2O_3	5.75	778	-	198

Figure 3.

AGING STUDIES WITH NO APPLIED STRESS

Aging studies with no applied stress were carried out on the four TTZ's to evaluate microstructural and phase stability, dimensional stability, and potential changes in room temperature K_{Ic}, MOR and MOE after exposure to varying times at temperature. The results are presented below:

MICROSTRUCTURAL STABILITY

Changes in the coarse level microstructure of TTZ's with MgO additions are evident after only 50 hours of aging at 1300°C. However, after even 250 hours at 1000°C there is little evidence of microstructural change. (See Figure 4). The microstructures shown in Figure 4 are typical of material F, C and MN. By contrast, the TTZ with the Y_2O_3 additive shows no evidence of microstructural change even after 250 hours at 1300°C. (See Figure 5). It should be noted that the fine level microstructure has not yet been examined and greater effects may be observed at the finer (i.e. 50-500Å) scale.

PHASE STABILITY

The phase stability of TTZ's was studied by measuring the changes in the percent of monoclinic phase as a function of both annealing temperature and time. The increase in the monoclinic phase is assumed to be the result of the spontaneous transformation of the metastable tetragonal phase, which provides the strength and toughness of the TTZ. Samples were annealed for 50, 100 and 250 hours for temperatures ranging between 900°C and 1300°C. Figure 6 shows the change in monoclinic phase with aging for two

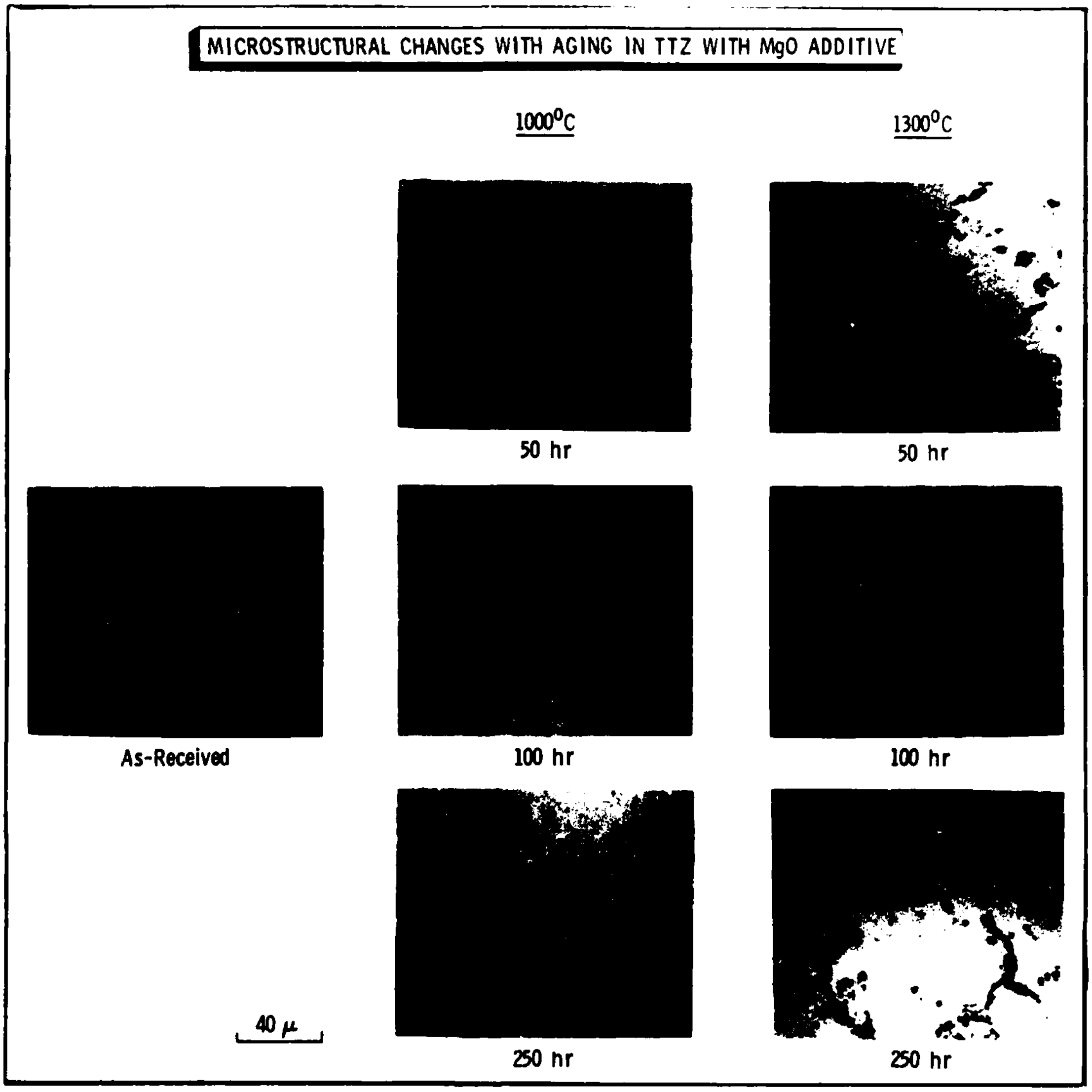


Figure 4.

MgO containing TTZ's and an Y_2O_3 containing TTZ. Both MgO containing TTZ's show similar behavior, namely the percent of monoclinic phase increases with time and temperature above 900°C. By contrast, the Y_2O_3 containing TTZ shows a relatively negligible change in the amount of monoclinic phase until approximately 1200°C-1300°C. Even at 1300°C, the Y_2O_3 containing TTZ has undergone less than half as much spontaneous transformation as the MgO containing materials.

DIMENSIONAL STABILITY

Dimensional stability was evaluated by measuring the change in density with aging time and temperature and is shown in Figure 7. The change in density results from three separate effects: a) increase in crystal lattice volume as a result of the tetragonal to monoclinic transformation, b) increasing porosity at grain boundaries (See Figure 4) and c) the presence of an additional (as yet unidentified) phase at the grain boundaries. Since it has been shown above that the MgO containing TTZ's have both a greater amount of tetragonal to monoclinic phase change, and a greater amount of grain boundary porosity on aging, it is not suprising that they exhibit significantly greater volume change than Y_2O_3 containing material.

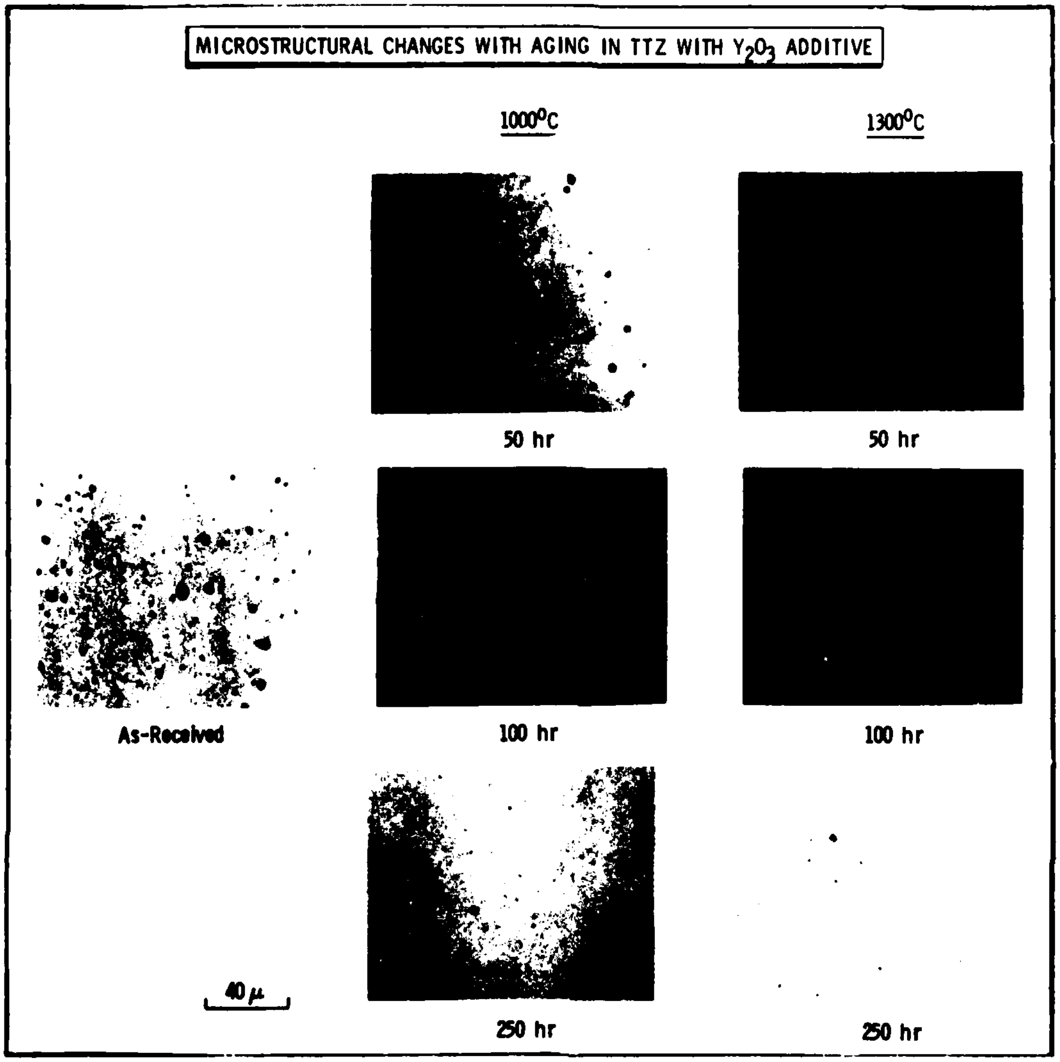


Figure 5.

RETENTION OF MECHANICAL PROPERTIES

Figure 8 shows the effect of aging on the room temperature mechanical properties for one MgO containing TTZ. This result is typical of several MgO containing TTZ's examined. The MOR tests were carried out as described in Reference (3). The test samples were generally .110 X .080 X 2 inch, tested in 4 pt. flexure with spans of 0.6 X 1.2 inch. The K_{IC} values were obtained by the indentation and fracture technique described by Lawn (4). MOE's were measured ultrasonically. It is significant that the retained RT mechanical properties all decrease with aging, with MOR showing the greatest change and MOE the least.

TIME-TEMPERATURE DEPENDANCE UNDER LOAD

Stepped Temperature Stress Rupture (STSR) testing (See Ref. 5) and limited supplemental standard stress rupture (SR) tests were carried out to obtain a preliminary evaluation of strength retention with time under load at various temperatures.

STEPPED TEMPERATURE STRESS RUPTURE TESTS

As shown in Figure 9. The STSR results for all three MgO containing TTZ's were essentially similiar. In this figure, the numbers shown above each curve are the stress in MPa and the numbers under the curve are the temperatures in °C.

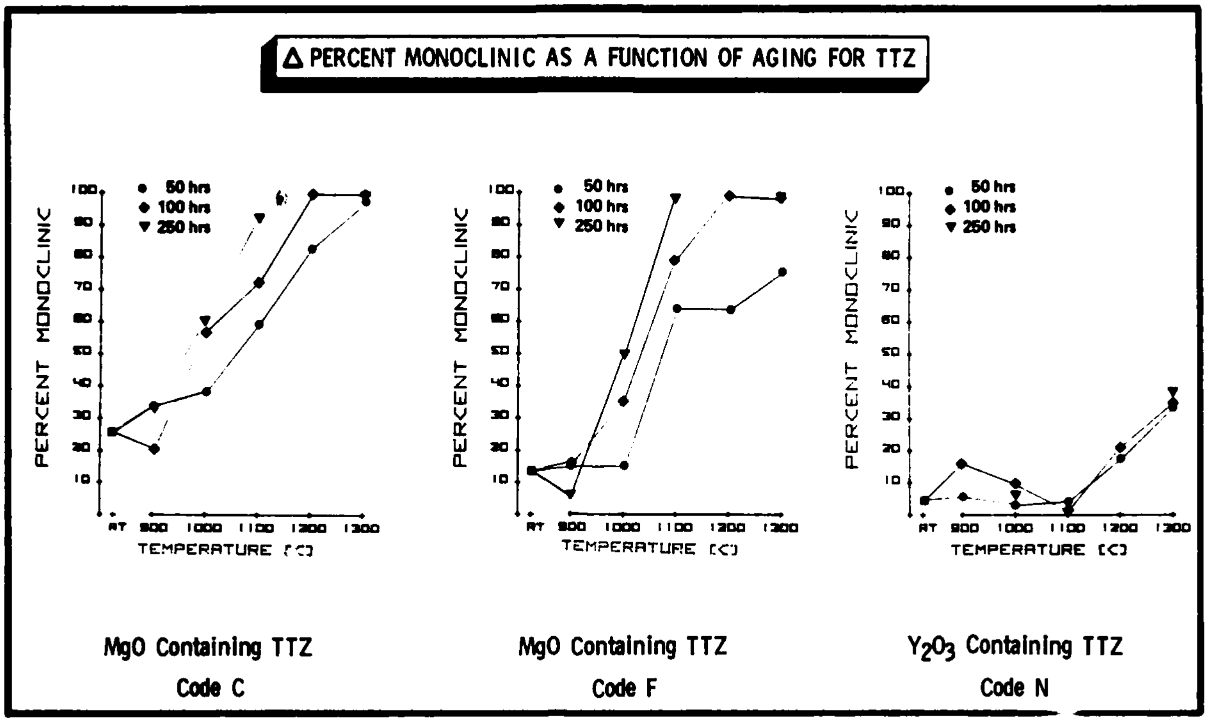


Figure 6.

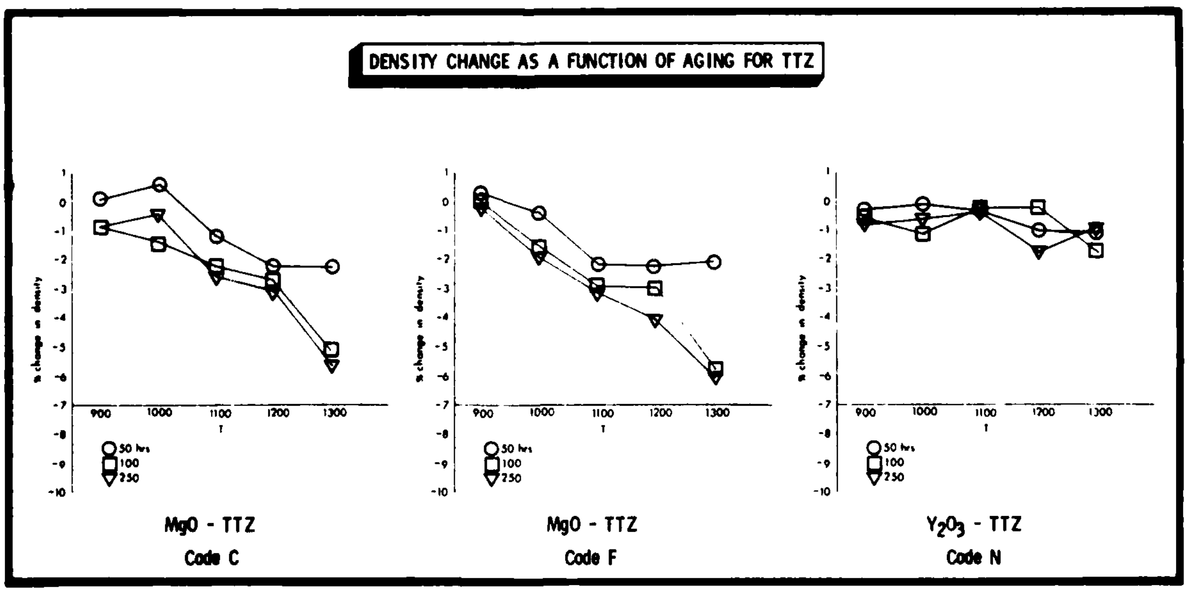


Figure 7.

EFFECT OF AGING ON PROPERTIES OF TTZ WITH MgO ADDITIONS

	RT MOR (MPa)	K_{IC} ($MN/m^{3/2}$)	MOE (GPa)
AS-RECEIVED	592	7.9	227
AGED 100 hr @ 1000°C	395	6.5	213
AGED 500 hr @ 1000°C	384	6.1	210

CODE C MATERIAL

Figure 8.

Each step on the curves represents a 24 hour interval. All samples were dead weight loaded in four point loading with 0.75 inch top and 1.50 inch bottom spans. Room temperature MOR's are shown to indicate the degree to which fracture strengths are reduced at elevated temperatures. No major evidence of static fatigue was encountered until 1200°C. However, in longer duration conventional stress-rupture testing, such behavior could well commence at lower temperatures. The failure mechanism for all of these MgO containing materials was via a creep fracture mode, with strains of 0.5 to 2.5%. In the case of material F the strains were large enough (up to 2.5%) so that the automatic shut off switch was tripped resulting in the premature test suspensions as shown on Figure 9. Creep strains of this magnitude resulted in the characteristic creep crack pattern shown in Figure 10.

In contrast, the STSR results for the Y_2O_3 containing TTZ, Figure 11, indicated that static fatigue occurred at all temperatures at which testing was carried out (800-1100°C). The mechanism appeared to be slow crack growth for temperatures at or below 1000°C, and creep fracture for temperatures above 1100°C. Where creep fracture was the predominant failure mode, high strains (0.4-0.8% at 1100°C) were encountered. It is rather surprising that material N, which showed the greatest stability of microstructure, phase composition and aid density, showed the greatest reduction in load carring capability with time and temperature. This may be due to the fact that this material contains 0.72 wt. % Si (present as SiO_2) which is believed to be present as a grain boundary glass (6,7). If the Si, and thus the glass, can be eliminated, then it is likely that Y_2O_3 containing TTZ's should show performance equal or superior to the MgO containing materials.

SUPPLEMENTAL STRESS RUPTURE TESTS

Sufficient samples of materials C and MN were available to conduct supplemental conventional SR tests. In addition, more time was accumulated in a SR test of an early varient of material C (herein refered to as C^1) which we reported on at last years Contractors Coordination Meeting (8). A summary of results from over 5200 hours of SR testing of eight tests bars is presented in Figure 12. Using this data, it was possible to estimate activation energies from creep in TTZ with MgO additives. (See Figure 13). Given the limited data available the activation energies of 75 Kcal/mole for material C and 100 Kcal/g mole for material MN are close enough that one can say they very likely share the same creep mechanism.

CONCLUSIONS

The conclusions of our studies to date indicate that:

1. All TTZ's investigated exhibit microstructural instabilities and degradation of strength at temperatures of 1000°C or less.

2. Microstructural and other evidence indicates that MgO containing TTZ looses its strength as the result of an overaging process.

3. SR testing has demonstrated that MgO containing TTZ's can sustain stresses of 30 Ksi for hundreds to thousands of hours at 900°C with small strains.

4. One variety of Y_2O_3 containing TTZ, which shows little microstructural evidence of aging, in fact has a greater time-temperature dependance of strength than the MgO containing TTZ's. Since this may be the consequence of a Si impurity (and resultant grain boundary glass), significant improvement of the high temperature mechanical properties of this material may be possible by elimination of the Si impurity.

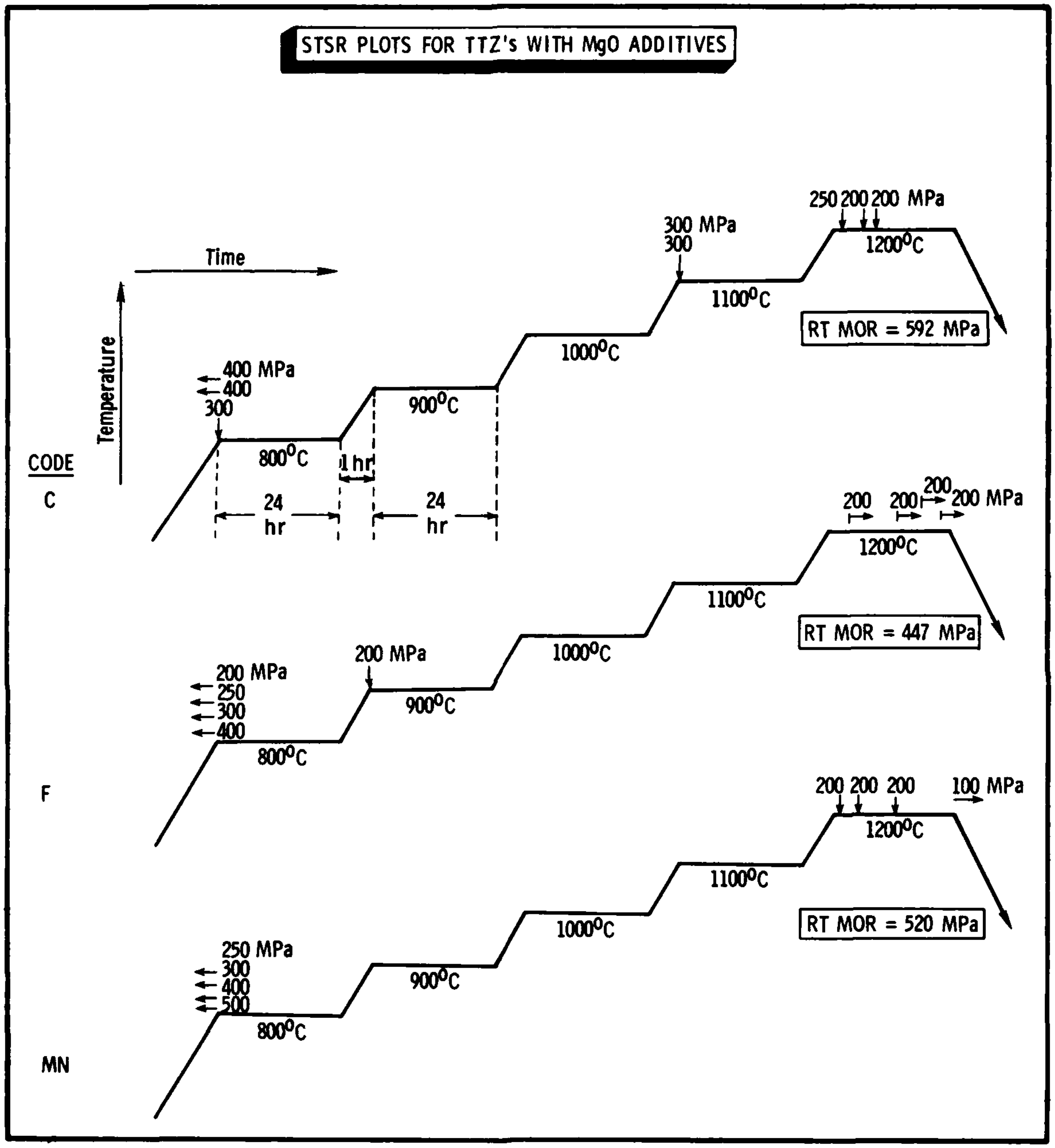
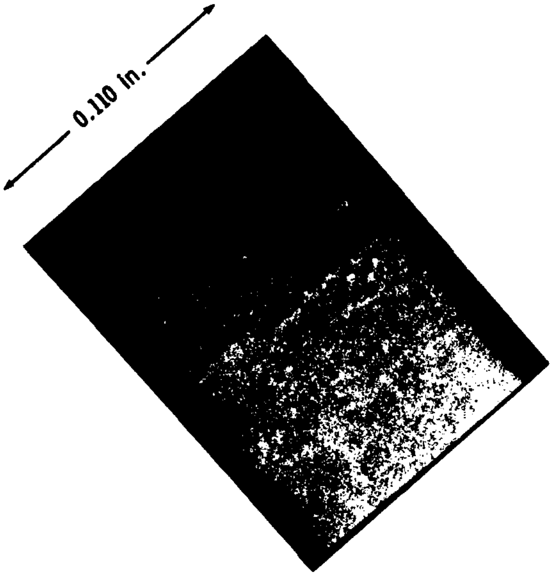

STSR PLOTS FOR TTZ's WITH MgO ADDITIVES
Time
Temperature
CODE
C
400 MPa
400
300
800°C
1 hr
24 hr
24 hr
900°C
1000°C
300 MPa
300
1100°C
250 200 200 MPa
1200°C
RT MOR = 592 MPa
F
200 MPa
250
300
400
800°C
200 MPa
900°C
1000°C
1100°C
200 200 200 200 MPa
1200°C
RT MOR = 447 MPa
MN
250 MPa
300
400
500
800°C
900°C
1000°C
1100°C
200 200 200 100 MPa
1200°C
RT MOR = 520 MPa

Figure 9.

TYPICAL PATTERN OF CREEP FRACTURE CRACKS ON TENSILE SURFACE OF MgO ADDITIVE TTZ



MATERIAL F, AFTER COMPLETION OF A STSR TEST INCLUDING THE FULL 24 HOURS AT 1200°C. TOTAL STRAIN CALCULATED FROM SPECIMEN CURVATURE, WAS 2.5%

Figure 10.

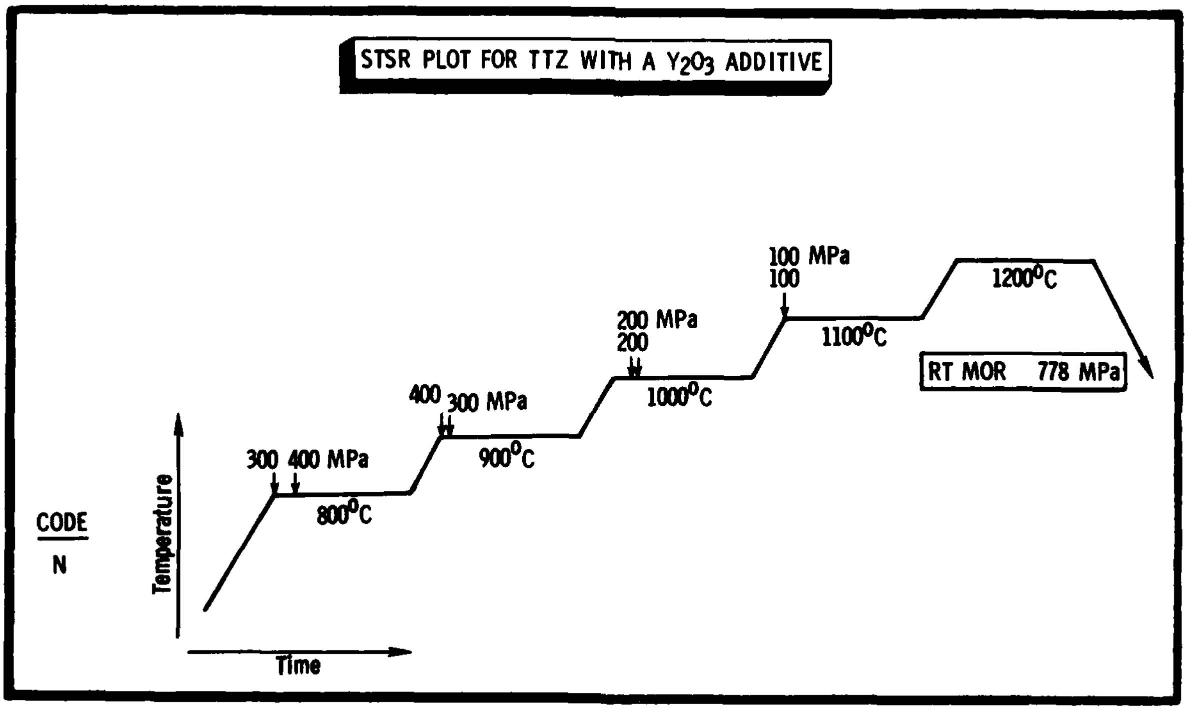


Figure 11.

STRESS-RUPTURE RESULTS OF TESTS ON MgO ADDITIVE TTZ's

CODE	STRESS (MPa)	TEMP. (°C)	TIME (hrs)	ϵ (%)	COMMENT
C	200	900	501+	0.04	SAMPLE SURVIVED. TEST TERMINATED
	200	1000	511+	0.23	SAMPLE SURVIVED, TEST TERMINATED
	200	1100	125	1.03	SPECIMEN DEFORMED SUFFICIENT TO TRIP MICROSWITCH - NO FAILURE
	200	1200	26	0.96	FAILURE
MN	200	900	502	NEG.	SAMPLE SURVIVED, TEST TERMINATED
	200	1000	502	0.58	SAMPLE SURVIVED. TEST TERMINATED
	200	1100	73	1.62	FAILURE
C'	200	900	3020	0.20	SAMPLE SURVIVED, MOR IN FAST FRACT AT RT - 55% OF AVE. AS-RECEIVED, RT MOR

Figure 12.

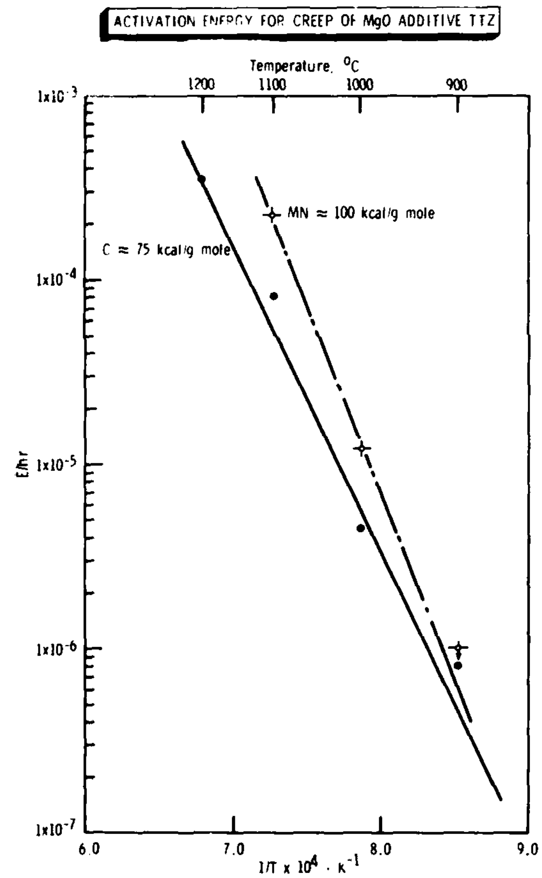


Figure 13.

ACKNOWLEDGMENT

This work was supported by the Department of Energy under an interagency agreement with AMMRC (DOE IA Noi DE-A101-77CS51017). The authors would also like to thank Drs. Brian Lawn and A. C. Gonzalez of the National Bureau of Standards for their assistance in obtaining the fracture toughness measurements.

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TIME-TEMPERATURE DEPENDENCE OF THE
STRENGTH OF COMMERCIAL ZIRCONIA CERAMICS -
Liselotte J. Schioler, George D. Quinn,
and R. Nathan Katz

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Technical Report AMMRC TR 84-16, April 1984, 11 pp - illus, Interagency Agreement DE-AE-101-77 CS51017, AMCMS Code 69200R.8891

The unusual combination of attractive properties exhibited by transformation toughened zirconias (TTZ's) has focused attention on them as leading candidates for application in the adiabatic Diesel engine. These materials are age-hardened ceramic alloy systems and as such they are likely to be susceptible to overaging and loss of strength at long times at high temperatures. This paper presents preliminary data on the microstructural, phase and dimensional stability of aged TTZ's together with some data on the effects of aging on strength, toughness and modulus. Stress rupture data on TTZ's with both MgO and Y_2O_3 additions show a considerable decrease in load carrying ability at temperatures of 800°C and above.

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